NATIONAL ENVIRONMENTAL LABORATORY ACCREDITATION CONFERENCE

DRAFT QUALITY SYSTEMS

June 1, 1996

QUALITY SYSTEMS

TABLE OF CONTENTS

5.0 QUALITY SYSTEMS

INTRODUCTION

Quality Systems include all quality assurance (QA) policies and quality control (QC) procedures, which shall be delineated in a QA Plan to help ensure and document the quality of the analytical data. Laboratories seeking accreditation under NELAP must assure implementation of all QA policies and the essential applicable QC procedures specified in this chapter. The QA policies, which establish essential QC procedures, are applicable to environmental laboratories regardless of size and complexity.

It is the intent of this section to provide sufficient detail concerning QA and QC requirements so that all accrediting authorities evaluate laboratories consistently and uniformly.

Section 5 is organized according to the structure of ISO/IEC Guide 25, 1990. The text from ISO/IEC Guide 25 is in bold and a different font. Modifications to this text have been made to eliminate confusion as to the intent or use of ISO/IEC Guide 25 language in the NELAP standards. In these cases, the changes are noted as underlined and in a different font for proposed text and struck through for deleted text. Where deemed necessary, specific areas within this section may contain more information than specified by ISO/IEC Guide 25.

All items identified in this chapter shall be available for on-site inspection or data audit.

5.1 SCOPE

a) **This** Standard ~~**Guide**~~ **sets out the general requirements in accordance with which a laboratory has to demonstrate that it operates, if it is to be recognized as competent to carry out specific** environmental ~~**calibrations or**~~ **tests.**

b) **Additional requirements and information which have to be disclosed for assessing competence or for determining compliance with other criteria may be specified by the organization or authority granting the recognition (or approval), depending upon the specific character of the task of the laboratory.**

In addition to the ISO/IEC Guide 25 standard, the supplemental language in this chapter, specifies the essential activities, records and procedures that a

laboratory must implement to be considered for accreditation under NELAP.

If more stringent standards or requirements are specified by the test method or by regulation, the laboratory shall demonstrate that such requirements are met.

c) **This** Standard ~~**Guide**~~ **is for use by** environmental ~~**calibration and**~~ **testing laboratories in the development and implementation of their quality systems. It may also be used by accreditation bodies, certification bodies and others concerned with the competence of** environmental **laboratories.**

5.2 REFERENCES

See Appendix A

5.3 DEFINITIONS

The relevant definitions from ISO/IEC Guide 2, ISO 8402, ANSI/ASQC E-4,1994, the EPA "Glossary of Quality Assurance Terms and Acronyms", **and the *International vocabulary of basic and general terms in metrology (VIM)* are applicable, the most relevant being quoted** in Appendix B ~~**below**~~ **together with further definitions applicable for the purposes of this** Standard ~~**Guide**~~**.**

See Appendix B

5.4 ORGANIZATION AND MANAGEMENT

5.4.1 Legal Definition of Laboratory

The laboratory shall be legally identifiable. It shall be organized and shall operate in such a way that its permanent, temporary and mobile facilities meet the requirements of this Standard ~~Guide~~.

5.4.2 Organization

The laboratory shall:

a) **have managerial staff with the authority and resources needed to discharge their duties;**

b) **have arrangements to ensure that its personnel are free from any commercial, financial and other pressures which might adversely affect**

the quality of their work;

c) **be organized in such a way that confidence in its independence of judgment and integrity is maintained at all times;**

d) **specify and document the responsibility, authority, and interrelation of all personnel who manage, perform or verify work affecting the quality of calibrations and tests;**

Such documentation shall include:

1) a clear description of the lines of responsibility in the laboratory and shall be proportioned such that adequate supervision is ensured. An organizational chart is recommended and

2) job descriptions for all positions.

e) **provide supervision by persons familiar with the calibration or test methods and procedures, the objective of the calibration or test and the assessment of the results. The ratio of supervisory to non-supervisory personnel shall be such as to ensure adequate supervision;**

f) **have a technical** director(s) ~~**manager**~~ **(however named) who has overall responsibility for the technical operations** ~~**s**~~ of the environmental testing laboratory**;**

The technical director shall certify that personnel with appropriate educational and/or technical background perform all tests for which the laboratory is certified. Such certification shall be documented.

The technical director shall meet the requirements specified in The Accreditation Process.

g) **have a quality** assurance officer ~~**manager**~~ **(however named) who has responsibility for the quality system and its implementation. The quality** assurance officer ~~**manager**~~ **shall have direct access to the highest level of management at which decisions are taken on laboratory policy or resources, and to the technical manager. In some laboratories, the quality** assurance officer ~~**manager**~~ **may also be the technical** director ~~**manager**~~ **or deputy technical** director ~~**manager**~~**;**

The quality assurance officer (and/or his/her designee) shall:

1) serve as the focal point for QA/QC and be responsible for the oversight and/or review of quality control data;

2) where applicable, have functions independent from laboratory operations for which they have quality assurance oversight;

3) be able to evaluate data objectively and perform assessments without outside (e.g., managerial) influence;

4) have documented training and/or experience in QA/QC procedures and be knowledgeable in the quality system as defined under NELAP;

5) have a general knowledge of the analytical methods for which data review is performed; and

6) where applicable, conduct internal audits on the entire technical operation annually.

h) **nominate deputies in case of absence of the technical** director **or quality** assurance officer ~~**manager**~~**;**
The laboratory shall accomplish this by having contingency plans in the event that either the technical director or quality assurance officer is absent.

i) **where relevant, have documented policy and procedures to ensure the protection of clients' confidential information and proprietary rights;**

j) **where appropriate, participate in inter-laboratory comparisons and proficiency testing programs.**

For purposes of qualifying for and maintaining accreditation, each laboratory shall participate in a proficiency test program as outlined in Chapter 2.0.

5.5 QUALITY SYSTEM - ESTABLISHMENT, AUDITS, ESSENTIAL QUALITY CONTROLS AND DATA VERIFICATION

5.5.1 Establishment

The laboratory shall establish and maintain a quality system appropriate to the type, range and volume of environmental ~~calibration and~~ testing activities it undertakes.

a. **The elements of this system shall be documented.**

b. **The quality documentation shall be available for use by the laboratory personnel.**

c. **The laboratory shall define and document its policies and objectives for, and its commitment to good laboratory practice and quality of ~~calibration or~~ testing services.**

d. **The laboratory management shall ensure that these policies and objectives are documented in a quality manual and communicated to, understood, and implemented by all laboratory personnel concerned.**

e. **The quality manual shall be maintained current under the responsibility of the quality assurance officer ~~manager~~.**

5.5.2 Quality Manual

The quality manual, and related quality documentation, shall state the laboratory's policies and operational procedures established in order to meet the requirements of this Standard ~~Guide~~.

The Quality Manual shall list on the title page: a document title; the laboratory's full name and address; the name, address (if different from above), and telephone number of individual(s) responsible for the laboratory; the name of the quality assurance officer (however named); the identification of all major organizational units which are to be covered by this quality manual and the effective date of the version;

The quality manual and related quality documentation shall also contain:

a) **a quality policy statement, including objectives and commitments, by top management;**

b) **the organization and management structure of the laboratory, its place in any parent organization and relevant organizational charts;**

c) **the relations between management, technical operations, support services and the quality system;**

d) **procedures for control and maintenance of documentation;** including document control of laboratory notebooks; instrument logbooks; standards logbooks; and records for data reduction, validation storage and reporting;

e) **job descriptions of key staff and reference to the job descriptions of other staff;**

f) **identification of the laboratory's approved signatories ~~(where this concept is appropriate)~~;** at a minimum, the title page must have the signed concurrence, (with appropriate titles) of all responsible parties including the QA officer, technical director, and laboratory owner (if applicable);

g) **the laboratory's procedures for achieving traceability of measurements;**

h) **the laboratory's scope of ~~calibrations and/or~~ tests;**

i) **ensuring that the laboratory reviews all new work to ensure that it has the appropriate facilities and resources before commencing such work;**

j) **reference to the calibration, verification and/or test procedures used;**

k) **procedures for handling** submitted samples **~~calibrations and test items~~;**

l) **reference to the major equipment and reference measurement standards used** as well as the facilities and services used by the laboratory**;**

m) **reference to procedures for calibration, verification and maintenance of equipment;**

n) **reference to verification practices including interlaboratory comparisons, proficiency testing programs, use of reference materials and internal quality control schemes;**

o) **procedures to be followed for feedback and corrective action whenever**

testing discrepancies are detected, or departures from documented policies and procedures occur;

p) **the laboratory management arrangements for exceptionally permitting departures from documented policies and procedures or from standard specifications;**

q) **procedures for dealing with complaints;**

r) **procedures for protecting confidentiality and proprietary rights;**

s) **procedures for audit and review;**

t) processes/procedures for establishing that personnel are adequately experienced in the duties they are expected to carry out and/or receive any needed training.

5.5.3 Audits

5.5.3.1 Internal Audits

The laboratory shall arrange for annual technical systems **audits of its activities at appropriate intervals to verify that its operations continue to comply with the requirements of the quality system. Such audits shall be carried out by** the quality assurance officer or designee(s) who are trained and qualified as auditors, and ~~**trained and qualified staff**~~ **who are, wherever possible, independent of the activity to be audited. Where the audit findings cast doubt on the correctness or validity of the laboratory's calibrations or test results, the laboratory shall take immediate corrective action and shall immediately notify, in writing, any client whose work may have been affected.**

5.5.3.2 Managerial Review

The quality system adopted to satisfy the requirements of this Standard ~~**Guide**~~ **shall be reviewed at least once a year by the management to ensure its continuing suitability and effectiveness and to introduce any necessary changes or improvements.**

5.5.3.3 Audit Review

All audit and review findings and any corrective actions that arise from them

shall be documented. The quality assurance officer ~~**person responsible for quality**~~ **shall ensure that these actions are discharged within the agreed timescale.**

5.5.3.4 Performance Audits

In addition to periodic audits the laboratory shall ensure the quality of results provided to clients by implementing checks. These checks shall be reviewed and shall include, as appropriate, but not be limited to:

a) **internal quality control schemes using whenever possible statistical techniques;** (see 5.5.4 below)

b) **participation in proficiency testing or other interlaboratory comparisons;**

c) ~~**regular**~~ **use of certified reference materials and/or in-house quality control using secondary reference materials** as specified in Section 5.5.4**;**

d) **replicate testings using the same or different methods;**

e) **re-testing of retained** samples ~~**items**~~**;**

f) **correlation of results for different characteristics of a**~~**n**~~ sample ~~**item**~~**.**

5.5.3.5 Corrective Actions

a) In addition to providing acceptance criteria and specific protocols for corrective actions in the Method Standard Operating Procedures (see 5.10.1.1), the laboratory shall implement general procedures to be followed to determine when quality control data are out of control. These procedures shall include but are not limited to the following:

   1) identify the individual(s) responsible for assessing each QC data type;

   2) identify the individual(s) responsible for initiating and/or recommending corrective actions;

   3) define how the analyst should treat a data set if the associated QC measurements are unacceptable;

4) specify how out-of-control situations and subsequent corrective actions are to be documented; and

5) specify procedures for management (including the QA officer) to review corrective action reports.

b) To the extent possible, samples shall be reported only if all quality control measures are acceptable. If a quality control measure is found to be out of control, and the data is to be reported, all samples associated with the failed quality control measure shall be reported with the appropriate data qualifying code(s).

5.5.4 Essential Quality Control Procedures

The following general quality control principles shall apply, where applicable, to all testing laboratories. The manner in which they are implemented is dependent on the types of tests performed by the laboratory (i.e., chemical, microbiological, radiological). The standards for any given test type shall assure that the applicable principles are addressed:

a) All laboratories shall have protocols (as required in Section 5.10.1.1) in place to monitor the following quality controls:

1) Adequate positive and negative controls to monitor tests such as blanks, spikes, reference toxicants, zero blanks;

2) Adequate tests to define the variability and/or reproducibility of the laboratory results such as duplicates;

3) Measures to ensure the accuracy of the test data including sufficient calibration and/or continuing calibrations, use of certified reference materials, proficiency test samples, or other measures;

4) Measures to evaluate test performance, such as method detection limits or range of applicability such as linearity;

5) Selection of appropriate formulae to reduce raw data to final results such as linear regression, internal standards, or statistical packages;

6) Selection and use of reagents and standards of appropriate quality;

7) Measures to assure the selectivity of the test for its intended purpose; and

8) Measures to assure constant and consistent test conditions (both instrumental and environmental) where required by the method such as temperature, humidity, light, or specific instrument conditions.

b) All quality control measures shall be assessed and evaluated on an on-going basis, and quality control acceptance limits shall be used to determine the validity of the data. The acceptance/rejection criteria shall be updated at a frequency established by the method or by the NELAP standards.

c) The laboratory shall have procedures for the development of acceptance/rejection criteria where no method or regulatory criteria exists.

d) The method specified and/or method-recommended quality control protocols shall be followed. The essential standards outlined in Appendix D shall be used if no protocols are written into the method or if the method protocols are less stringent.

The essential quality control measures for testing categories are found in Appendix D of this chapter.

5.6 PERSONNEL

5.6.1 General requirements for laboratory staff

The laboratory shall have sufficient personnel, having the necessary education, training, technical knowledge and experience for their assigned functions.

All personnel shall be responsible for complying with all quality assurance/quality control requirements that pertain to their organizational/technical function. Each laboratory staff member must have a combination of experience and education to demonstrate adequately a specific knowledge of their particular function and a general knowledge of laboratory operations, analytical methods, quality assurance/quality control procedures and records management.

5.6.2 Laboratory Management Responsibilities

Laboratory management shall be responsible for:

a) defining the minimal level of qualification, experience and skills necessary for all positions in the laboratory. In addition to education and/or experience, basic laboratory skills such as using a balance, colony counting, aseptic techniques or chemically transferring reagents shall be considered;

b) Assuring that all technical laboratory staff have demonstrated initial and ongoing proficiency in the activities for which they are responsible. Such demonstration shall be documented;

c) ~~**The laboratory shall**~~ Ensuring ~~**ensure**~~ **that the training of its personnel is kept up-to-date;**
Training shall be considered up-to-date when documentation in the files indicate acceptable performance of a blind sample at least once per year and a signature certifying that technical personnel have read, understood and agreed to perform the most recent version of the method, the approved method (if applicable) or standard operating procedure. Evidence must be on file that demonstrates all employees are aware and using the latest edition of the laboratories* in house quality documentation. Training courses or workshops on specific equipment, analytical techniques or laboratory procedures shall all be documented;

d) Documenting all analytical and operational activities of the laboratory;

e) Supervision of all personnel employed by the laboratory, including those persons designated as principle analysts;

f) Assuring that all sample acceptance criteria (Section 5.9) are met and that samples are logged into the sample tracking system and properly labeled and stored; and

g) The production and quality of all data reported by the laboratory.

5.6.3 Records

Records on the relevant qualifications, training, skills and experience of the technical personnel shall be maintained by the laboratory, including records on demonstrated proficiency for each laboratory method, such as the criteria outlined in 5.10.2.1 for chemical testing.

5.7 PHYSICAL FACILITIES - ACCOMMODATION AND ENVIRONMENT

5.7.1 Environment

a) **Laboratory accommodation, ~~calibration and~~ test areas, energy sources, lighting, heating and ventilation shall be such as to facilitate proper performance of ~~calibrations or~~ tests.**

b) **The environment in which these activities are undertaken shall not invalidate the results or adversely affect the required accuracy of measurement. Particular care shall be taken when such activities are undertaken at sites other than the permanent laboratory premises.**

c) **The laboratory shall provide facilities for the effective monitoring, control and recording of environmental conditions as appropriate. ~~Due~~ Attention shall be paid, for example, to biological sterility, dust, electromagnetic interference, humidity, mains voltage, temperature, and sound and vibration levels, as appropriate to the calibrations or tests concerned.**

d) In instances where monitoring or control of any of the above mentioned items are specified in a test method or by regulation, the laboratory shall meet and document adherence to the laboratory facility requirements.

NOTE - It is the laboratory's responsibility to comply with the relevant health and safety requirements. This aspect, however, is outside the scope of this Guide.

5.7.2 Work Areas

a) **There shall be effective separation between neighboring areas when the activities therein are incompatible** including culture handling or incubation areas and volatile organic chemicals handling areas.

b) **Access to and use of all areas affecting the quality of these activities**

shall be defined and controlled.

c) **Adequate measures shall be taken to ensure good housekeeping in the laboratory** and to assure that contamination is unlikely.

d) Work spaces must be available to ensure an unencumbered work area. Work areas include:

1) access and entryways to the laboratory;
2) sample receipt area(s);
3) sample storage area(s);
4) chemical and waste storage area(s); and
5) data handling and storage area(s).

5.8 EQUIPMENT AND REFERENCE MATERIALS

a) **The laboratory shall be furnished with all items of equipment (including reference materials) required for the correct performance of ~~calibrations and~~ tests** for which accreditation is sought**. In those cases where the laboratory needs to use equipment outside its permanent control it shall ensure that the relevant requirements of this** Standard **~~Guide~~ are met.**

b) **All equipment shall be properly maintained,** inspected and cleaned**. Maintenance procedures shall be documented.**

c) **Any item of the equipment which has been subjected to overloading or mishandling, or which gives suspect results, or has been shown by verification or otherwise to be defective, shall be taken out of service, clearly identified and wherever possible stored at a specified place until it has been repaired and shown by calibration, verification or test to perform satisfactorily. The laboratory shall examine the effect of this defect on previous** calibrations or **tests.**

d) **Each item of equipment including reference materials shall, when appropriate, be labeled, marked or otherwise identified to indicate its calibration status.**

e) **Records shall be maintained of each major item of equipment and all reference materials significant to the ~~calibrations or~~** analytical **tests performed.** These records shall include documentation on

all routine and nonroutine maintenance activities and reference material verifications.

The records shall include:

1) **the name of the item of equipment;**
2) **the manufacturer's name, type identification, and serial number or other unique identification;**
3) **date received and date placed in service;**
4) **current location, where appropriate;**
5) **condition when received (e.g. new, used, reconditioned);**
6) **copy of the manufacturer's instructions, where available;**
7) **dates and results of calibrations and/or verifications and date of the next calibration and/or verification;**
8) **details of maintenance carried out to date and planned for the future; and**
9) **history of any damage, malfunction, modification or repair.**

5.9 MEASUREMENT TRACEABILITY AND CALIBRATION

5.9.1 General Requirements

All measuring and testing equipment having an effect on the accuracy or validity of ~~calibrations or~~ tests shall be calibrated and/or verified before being put into service. The laboratory shall have an established program for the calibration and verification of its measuring and test equipment. This includes balances, thermometers and control standards. Test equipment and measuring operations shall be standardized and verified before use and on a continuing basis.

5.9.2 Traceability of Calibration

a) **The overall program of calibration and/or verification and validation of equipment shall be designed and operated so as to ensure that, wherever applicable, measurements made by the laboratory are traceable to national standards of measurement where available.**

b) **Calibration certificates shall wherever applicable indicate the traceability to national standards of measurement and shall provide the measurement results and associated uncertainty of measurement and/or a statement of compliance with an identified metrological specification.** The laboratory shall maintain records of all such

certifications.

c) **Where traceability to national standards of measurement is not applicable, the laboratory shall provide satisfactory evidence of correlation of results, for example by participation in a suitable program of interlaboratory comparisons or proficiency testing.**

5.9.3 Reference Standards

a) **Reference standards of measurement held by the laboratory shall be used for calibration only and for no other purpose, unless it can be demonstrated that their performance as reference standards has not been invalidated. Reference standards of measurement shall be calibrated by a body that can provide traceability to a national standard of measurement.**

b) **There shall be a program of calibration and verification for reference standards.**

c) **Where relevant, reference standards and measuring and testing equipment shall be subjected to in-service checks between calibrations and verifications. Reference materials shall, where possible, be traceable to national or international standards of measurement, or to national or international standard reference materials.**

5.9.4 Documentation and Labeling of Standards and Reagents

a) The laboratory shall retain records, such as manufacturer's statement of purity, of the origin, purity and traceability of all standards and reagents (including balance weights and thermometers). These records shall include the date of receipt, storage conditions, and, if applicable, the date of opening and an expiration date.

b) Detailed records shall be maintained on reagent and standard preparation. These records shall indicate traceability to purchase stocks or neat compounds, and must include the date of preparation and preparer's initials.

c) Where calibrations do not include the generation of a calibration curve, such as thermometers, balances, or titrations, records shall indicate the calibration date

and type (balance weight, thermometer serial number, primary standard concentration) of calibration standard that was used.

d) All prepared reagents and standards must be uniquely identified and the contents shall be clearly identified with preparation date, concentration(s) and preparer's initials.

5.9.5 Calibration

5.9.5.1 General Requirements

a) All calibration curves shall be dated and labeled with method, instrument, analysis date, analyte concentrations and analyte response (or response factor).

b) When used, the axes of the calibration curve shall be labeled. For electronic data processing systems that automatically compute the calibration curve, the equation for the curve and the correlation coefficient must be recorded. The equation for the line and the correlation coefficient shall also be recorded when the calibration curve is prepared manually.

c) A criteria for the acceptance of a calibration curve, for example, an acceptable correlation coefficient, shall be established and documented. If applicable, the method specified criteria shall be met.

5.9.5.2 Acceptance Criteria for Support Equipment

5.9.5.2.1 Analytical Support Equipment

These standards apply to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, temperature measuring devices (including thermometers and thermistors) and volumetric dispensing devices (such as Repipet®, Eppendorf®, or automatic dilutor/dispensing devices). All support equipment shall be:

a) maintained in proper working order following the maintenance schedule recommended by the manufacturer. The records of all activities including service calls shall be

kept.

b) calibrated annually, using NIST traceable references, over the entire range in which the equipment is used. The results of such calibration shall be within ± the manufacturer's stated sensitivity or:

1) The equipment shall be removed from service until repaired;

or

2) The laboratory shall prepare a deviation curve and correct all measurements for the deviation.

All measurements shall be recorded and maintained.

c) Prior to each day's use, balances, ovens, refrigerators, freezers, incubators and water baths shall be checked with NIST traceable references in the expected use range. Additional monitoring as prescribed by the method shall be performed for any device that is used in a critical test (such as incubators or water baths). The acceptability of use or continued use shall be per method specified requirements or ± the manufacturer's stated sensitivity.

5.9.5.2.2 Autoclaves

The sterilization temperature and pressure of each run must be documented by the use of appropriate chemical or biological sterilization indicators. Autoclave tape may be used to indicate that a load has been processed, but not to demonstrate completion of an acceptable sterilization cycle.

5.9.5.3 Initial Calibrations

a) When available, all initial calibrations shall be verified with standards obtained from a second or different source. These verification standards shall be analyzed with each initial calibration.

b) Calibration curves shall be prepared as specified in the method. If a method does not provide guidance in the preparation of a calibration curve, the laboratory shall establish the appropriate number of standards for use in the initial calibration using the following:

1) Determine the percent relative standard deviation (%RSD) by
   a. Taking at least seven replicate measurements of a standard with a concentration approaching the lowest quantitation level or;
   b. Performing a calibration linearity test (such as response factor or calibration factor) on at least 3 standards having concentrations that cover over the expected calibration range.
2) The minimum number of standards to be used in the initial calibration is dependent on the resulting %RSD:

%RSD	Number of Calibration Points
0 - <2	1**
2 - <10	3
10 - <25	5
>25	7

**Assumes linearity through the origin (0.0). For analytes for which there is no origin (such as pH), a two point calibration curve shall be used.

4) If the resulting curve is non-linear, additional standards shall be used.

3) The number of standards as determined from the above table and a blank shall be used for the initial calibration of the method.

c) The sample results must be bracketed by calibration standards under all circumstances. In those situations where the result will be used in a decision related to the

determination of a non-occurrence or “non-detect” of a contaminant, the standard shall approach the lowest quantitative level for the method.

d) In addition to the verification by second-source standards [see a) above], the calibration curve shall be subjected to a calibration linearity test, such as a linear regression or percent RSD.

5.9.5.4 Calibration Verification

When not specified by the analytical method, the value of the analyte(s) in the following calibration verification standards shall be within 15% of the true value unless the laboratory can demonstrate that wider limits are applicable.

5.9.5.4.1 Initial Calibration Verification

a) When an initial calibration curve is not run on the day of analysis, the integrity of the initial calibration curve shall be verified on each day of use (or 24 hour period) by initially analyzing a blank and a standard at the method specified concentration or a mid-level concentration if not specified by method.

b) If the initial calibration verification fails, the standard shall be immediately analyzed and evaluated a second time. If the results are still unacceptable, a new initial calibration curve shall be established and verified.

5.9.5.4.2 Continuing Calibration Verification

Additional standards shall be analyzed after the initial calibration curve or the integrity of the initial calibration curve (see 5.9.5.3.a or 5.9.5.4.1 above) has been accepted.

a) These standards shall be analyzed at a frequency of 5% or every 12 hours whichever is more frequent and may be the standards used in the original calibration curve or standards from another source. The frequency shall be increased if the instrument consistently drifts outside acceptable limits before the next calibration.

b) The concentration of these standards shall be determined by the anticipated or known concentration of the samples and/or method specified levels. At least one standard

shall be at a low level concentration. To the extent possible, the samples in each interval (i.e. every 20 samples or every 12 hours) should be bracketed with standard concentrations closely representing the lower and upper range of reported sample concentrations. If this is not possible, the standard calibration checks should vary in concentration throughout the range of the data being acquired.

c) A new curve shall be run if two back-to-back runs of one continuing calibration check is outside acceptable limits. When the continuing calibration check limit is exceeded high (i.e., high bias), and there are non-detects for the corresponding analyte in all environmental samples associated with the continuing calibration check, then those non-detects may be reported otherwise, the samples affected by the unacceptable check shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Additional sample analysis cannot occur until a new calibration curve is established and verified.

5.10 TEST METHODS AND STANDARD OPERATING PROCEDURES

5.10.1 Methods Documentation

a) **The laboratory shall have documented instructions on the use and operation of all relevant equipment, on the handling and preparation of** samples ~~**items**~~ **and for calibration and/or testing, where the absence of such instructions could jeopardize the calibrations or tests.**

b) **All instructions, standards, manuals and reference data relevant to the work of the laboratory shall be maintained up-to-date and be readily available to the staff.**

5.10.1.1 Standard Operating Procedures (SOPs)

Laboratories shall maintain standard operating procedures that accurately reflect all phases of current laboratory activities such as assessing data integrity, corrective actions, handling customer complaints, and all test methods.

a) These documents may be equipment manuals provided by the manufacturer, or internally written documents.

b) The test methods may be copies of published methods as long as any changes in the methods are documented and included in the methods manual (see 5.10.1.2).

c) Copies of all SOPs shall be accessible to all personnel.

d) The SOPs shall be logically organized and shall have the signature(s) of the approving authority.

e) Each SOP shall clearly indicate the effective date of the document, and the revision number.

5.10.1.2 Laboratory Method Manual(s)

a) The laboratory shall have and maintain an in-house methods manual(s) for each analyte or test to be certified.

b) This manual may consists of copies of published or referenced methods or standard operating procedures that have been written by the laboratory. Each method shall include where applicable:

1) identification of the test method and where applicable, the analyte name with qualifier (the qualifier is a word, phrase or number that better identifies the method; e.g., "Iron, Total", or "Chloride, Automated Ferricyanide", or "Our Lab. Method SOP No. 101");
2) applicable matrix or matrices;
3) method detection limit;
4) scope and application;
5) summary of the method;
6) definitions;
7) interferences;
8) safety;
9) equipment and supplies;
10) reagents and standards;
11) sample collection, preservation, shipment and storage;
12) quality control;
13) calibration and standardization;
14) procedure;
15) calculations;
16) method performance;
17) pollution prevention;
18) data assessment and acceptance criteria for quality control measures;

19) corrective actions for out-of-control data;
20) contingencies for handling out-of-control or unacceptable data;
21) waste management;
22) references; and
23) any tables, diagrams, flowcharts and validation data

c) In cases where minor modifications to the published method have been made by the laboratory such as change in type of column or change in operating conditions, or where the referenced method is ambiguous or provides insufficient detail such as reagent purity or reagent concentration, these changes or clarifications shall be documented as an appendix to the referenced method.

5.10.2 Test Methods

a) **The laboratory shall use appropriate methods and procedures for all ~~calibrations and~~ tests and related activities within its responsibility (including sampling, handling, transport and storage, preparation of items, estimation of uncertainty of measurement and analysis of ~~calibration and/or~~ test data). They shall be consistent with the accuracy required, and with any standard specifications relevant to the calibrations or tests concerned.**

b) When the use of mandated methods for a sample matrix is required, only those methods shall be used unless an EPA program office allows for the use of performance-based methods or non-legally mandated methods. In these cases, the laboratory shall meet the relevant start-up and ongoing validation procedures and calibrations as specified in 5.10.2.1 including a method detection limit study (D.1.4.a.).

c) **Where methods are not specified, the laboratory shall, wherever possible, select methods that have been published in international or national standards, those published by reputable technical organizations or in relevant scientific texts or journals.**

d) **Where it is necessary to employ methods that have not been established as standard, these shall be subject to agreement with the** accrediting authority **and client, be fully documented and validated, and be available to the client and other recipients of the relevant reports.**

e) The criteria listed in 5.10.2 b must be met for all methods.

5.10.2.1 Method Validation/Initial Demonstration of Method Performance

Prior to acceptance and institution of any method, satisfactory initial demonstration of method performance, in conformance with the relevant EPA guidelines, is required. In the absence of method-specified requirements, this demonstration shall follow the outlined protocols in Appendix C of this document. Thereafter, continuing demonstration of method performance, in conformance with the relevant EPA guidelines, is required. In both cases, the appropriate standard performance checklist (see Appendix C) must be completed and retained by the laboratory to be made available upon request. All associated supporting data

necessary to reproduce the analytical results summarized in the checklists must be retained by the laboratory. Initial demonstration of method performance must be completed each time there is a change in equipment, personnel or procedure.

5.10.3 Sample Aliquots

Where sampling (as in obtaining sample aliquots from a submitted sample) **is carried out as part of the test method, the laboratory shall use documented procedures and appropriate statistical techniques to** obtain representative subsamples. ~~**select samples**~~**.**

5.10.4 Data Verification

Calculations and data transfers shall be subject to appropriate checks.

a) The laboratory shall establish Standard Operating Procedures to ensure that the reported data is free from transcription and calculation errors.

b) The laboratory shall establish a Standard Operating Procedures to ensure that all quality control measures are reviewed, and evaluated before data is reported.

5.10.5 Documentation and Labeling of Standards and Reagents

Documented procedures shall exist for the purchase, reception and storage of consumable materials used for the technical operations of the laboratory.

a) The laboratory shall retain records, such as manufacturer's statement of purity, of the origin, purity and traceability of all standards and reagents (including balance weights and thermometers). These records shall include the date of receipt, storage conditions, and, if applicable, the date of opening and an expiration date.

b) Detailed records shall be maintained on reagent and standard preparation. These records shall indicate traceability to purchase stocks or neat compounds, and must include the date of preparation and preparer's initials.

c) Where calibrations do not include the generation of a calibration curve, such as thermometers, balances, or titrations, records shall indicate the calibration date

and type (balance weight, thermometer serial number, primary standard concentration) of calibration standard that was used.

d) All prepared reagents and standards must be uniquely identified and the contents shall be clearly identified with preparation date, concentration(s) and preparer's initials.

5.10.6 Computers and Electronic Data Related Requirements

Where computers or automated equipment are used for the capture, processing, manipulation, recording, reporting, storage or retrieval of ~~calibration or~~ test data, the laboratory shall ensure that:

a) **all requirements of this Standard ~~Guide~~ are complied with.** Section 8.1 through 8.11 of the EPA Document "2185 - Good Automated Laboratory Practices" (1995), shall be adopted as the standard for all laboratories employing microprocessors and computers.

b) **computer software is documented and adequate for use;**

c) **procedures are established and implemented for protecting the integrity of data; such procedures shall include, but not be limited to, integrity of data entry or capture, data storage, data transmission and data processing;**

d) **computer and automated equipment is maintained to ensure proper functioning and provided with the environmental and operating conditions necessary to maintain the integrity of calibration and test data;**

e) **it establishes and implements appropriate procedures for the maintenance of security of data including the prevention of unauthorized access to, and the unauthorized amendment of, computer records.**

5.11 SAMPLE HANDLING, SAMPLE ACCEPTANCE POLICY AND SAMPLE RECEIPT

Regardless of the laboratory's level of control over sampling activities, the following are essential to ensure sample integrity and valid data.

5.11.1 Sample Tracking

a) **The laboratory shall have a documented system for uniquely identifying the items to be ~~calibrated or~~ tested, to ensure that there can be no confusion regarding the identity of such items at any time.** This system shall include identification for all samples, subsamples and subsequent extracts and/or digestates. The laboratory shall assign a unique identification (ID) code

to each sample container received in the laboratory. Multiple aliquots of a sample that have been received for different analytical tests, such as nutrients, metals, or VOCs, must be assigned a different ID code, such as a prefix or suffix. The use of container shape, size or other physical characteristic, such as amber glass, or purple top, is not an acceptable means of identifying the sample.

b) This laboratory code shall maintain an unequivocal link with the unique field ID code assigned each container.

c) The laboratory ID code shall be placed on the sample container as a durable label.

d) The laboratory ID code shall be entered into the laboratory records (see 5.11.3.d) and shall be the link that associates the sample with related laboratory activities such as sample preparation or calibration.

e) In cases where the sample collector and analyst are the same individual or the laboratory preassigns numbers to sample containers, the laboratory ID code may be the same as the field ID code.

5.11.2 Sample Acceptance Policy

The laboratory shall have a written sample acceptance policy that clearly outlines the circumstances under which samples will be accepted. Data from any samples which do not meet the following criteria must be flagged in an unambiguous manner clearly defining the nature and substance of the variation. This sample acceptance policy shall be made available to sample collecting personnel and shall include, but is not limited to, the following areas of concern:

a) Proper, full, and complete documentation, which shall include sample identification, the location, date and time of collection, collector's name, preservation type, sample type and any special remarks concerning the sample;

b) Proper sample labeling to include unique identification and a labeling system for the samples with requirements concerning the durability of the labels (water resistant) and the use of indelible ink;

c) Use of appropriate sample containers.

d) Adherence to specified holding times; and

e) Adequate sample volume. Sufficient sample volume must be available to perform the necessary tests.

5.11.3 Sample Receipt Protocols

a) **Upon receipt, the condition of the** sample ~~**calibration or test item**~~**, including any abnormalities or departures from standard condition as prescribed in the relevant** ~~**calibration or**~~ **test method, shall be recorded.** All items specified in 5.11.2 above shall be checked.

1) All samples which require thermal preservation shall be considered acceptable if the arrival temperature is within +/-2°C of the required temperature or the method specified range. For samples with a specified temperature of 4°C, samples with a temperature of 0.1 to 6°C shall be acceptable. Samples that are hand delivered to the laboratory immediately after collection may not meet this criteria. In these cases, the samples shall be considered acceptable if there is evidence that the chilling process has begun such as arrival on ice.

2) The laboratory SOP shall define the procedures for checking chemical preservation using readily available techniques, such as pH, free chlorine or temperature, prior to sample preparation or analysis.

b) The results of all checks shall be recorded.

c) **Where there is any doubt as to the item's suitability for** testing**,** ~~**calibration or test**~~**, where the** sample ~~**item**~~ **does not conform to the description provided, or where the** ~~**calibration or**~~ **test required is not fully specified, the laboratory shall consult the client for further instruction before proceeding. The laboratory shall establish whether the** sample ~~**item**~~ **has received all necessary preparation, or whether the client requires preparation to be undertaken or arranged by the laboratory.** If the sample does not meet the sample receipt acceptance criteria listed in 5.11.3.a, 5.11.3.b or 5.11.3.c, the laboratory shall either:

1) Retain ~~all~~ correspondence and/or records of conversations concerning the final disposition of

rejected samples; or

2) Fully document any decision to proceed with the analysis of compromised samples:

i. The condition of these samples shall, at a minimum, be noted on the chain of custody or transmittal form and laboratory receipt documents.

ii. The analysis data shall be appropriately "qualified" on the final report.

d) The laboratory shall utilize a permanent, sequential log, such as a log book or electronic record, to document receipt of all sample containers. The following information must be recorded in the laboratory chronological log:

1) Date and time of laboratory receipt of sample;
2) Sample collection date;
3) Unique laboratory ID code (see 5.11.1);
4) Field ID code supplied by sample submitter;
5) Requested analyses, including approved method number, if applicable;
6) Signature or initials of data logger;
7) Comments resulting from inspection for sample acceptance rejection; and
8) Sampling kit code (if applicable).

e) All documentation, such as memos or transmittal forms, that is transmitted to the laboratory by the sample transmitter shall be retained.

f) A complete chain of custody record (Section 5.12.4), if utilized, shall be maintained.

5.11.4 Storage Conditions

The laboratory shall have documented procedures and appropriate facilities to avoid deterioration or damage to the sample ~~calibration or test item~~, during storage, handling, preparation, and ~~calibration or~~ test; any relevant instructions provided with the item shall be followed. Where items have to be stored or conditioned under specific environmental conditions, these conditions shall be maintained, monitored and recorded where necessary.

a) Samples shall be stored according to the conditions specified by preservation protocols:

1) Samples which require thermal preservation shall be stored under refrigeration which is +/- 2° of the specified preservation temperature unless method specific criteria exist. For samples with a specified temperature of 4°C, samples with a temperature of 0.1 to 6°C shall be acceptable.

2) Samples shall be stored away from all standards, reagents, food and other potentially contaminating sources.

b) Sample fractions, extracts, leachates and other sample preparation products shall be stored according to 5.11.4.a above or according to specifications in the method.

c. **Where a** sample ~~**calibration or test item**~~ **or portion of** the sample ~~**an item**~~ **is to be held secure (for example, for reasons of record, safety or value, or to enable check calibrations or tests to be performed later), the laboratory shall have storage and security arrangements that protect the condition and integrity of the secured items or portions concerned.**

5.11.5 Sample Disposal

The laboratory shall have documented procedures for the receipt, retention or safe disposal of calibration or test items, including all provisions necessary to protect the integrity of the laboratory.

5.12 RECORDS

The laboratory shall maintain a record system to suit its particular circumstances and comply with any applicable regulations. The system shall produce unequivocal, accurate records which document all laboratory activities. The laboratory ~~**It**~~ **shall retain on record all original observations, calculations and derived data, calibration records and a copy of the** ~~**calibration certificate, test certificate, or**~~ **test report for an appropriate period.**

There are two levels of record keeping: 1) sample custody or tracking and 2) legal or evidentiary chain of custody. All

essential requirements for sample custody are outlined in Sections 5.12.1, 5.12.2 and 5.12.3. The basic requirements for legal chain of custody (if required or implemented) are specified in Section 5.12.4.

5.12.1 Record Keeping System and Design

The records for each ~~calibration and~~ test shall contain sufficient information to permit ~~their~~ repetition. The record keeping system must allow historical reconstruction of all laboratory activities that produced the resultant sample analytical data. The history of the sample must be readily understood through the documentation. This shall include interlaboratory transfers of samples and/or extracts.

a) **The records shall include the identity of personnel involved in sampling, preparation, calibration or testing.**

b) All information relating to the laboratory facilities equipment, analytical methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification shall be documented.

c) The record keeping system shall facilitate the retrieval of all working files and archived records for inspection and verification purposes.

d) All documentation entries shall be signed or initialed by responsible staff. The reason for the signature or initials shall be clearly indicated in the records such as “sampled by”, “prepared by”, or “reviewed by”).

e) All generated data except those that are generated by automated data collection systems, shall be recorded directly, promptly and legibly in permanent ink.

f) Entries in records shall not be obliterated by methods such as erasures, overwritten files or markings. All corrections to record-keeping errors shall be made by one line marked through the error. The individual making the correction shall sign (or initial) and date the correction. These criteria also shall apply to electronically maintained records.

g) Refer to 5.10.6 for Computer and Electronic Data.

5.12.2 Records Management and Storage

a) **All records (including those pertaining to calibration and test equipment), certificates and reports shall be safely stored, held secure and in confidence to the client**. NELAC-related records shall be available to the accrediting authority.

b) All records of an organization that are pertinent to a specified project shall be retained for a minimum of five years unless otherwise designated for a longer period of time in another regulation. The records specified in 5.12.3 and 5.12.4 above shall be retained. All hardware and software necessary for the historical reconstruction of data must be maintained by the laboratory.

c) Records that are stored or generated by computers or personal computers (PCS) shall have hard copy or write-protected backup copies.

d) The laboratory shall establish a document control system which ensures that all standard operating procedures, manuals, or documents clearly indicate the time period during which the procedure or document was in force.

f) Access to archived information shall be documented with an access log. These records shall be protected against fire, theft, loss, environmental deterioration, vermin and, in the case of electronic records, electronic or magnetic sources.

h) In the event that a laboratory transfers or goes out of business, the laboratory shall have a plan to ensure that the records are maintained or transferred according to the clients' instructions.

5.12.3 Laboratory Sample Tracking

5.12.3.1 Sample Handling

A record of all procedures to which a sample is subjected while in the possession of the laboratory shall be maintained. These shall include but are not limited to all records pertaining to:

a) Sample preservation including appropriate sample container and compliance with holding time;
b) Sample identification, receipt, acceptance or rejection

and log-in;
c) Sample storage and tracking including shipping receipts, transmittal forms, and internal routing and assignment records;
d) Sample preparation including cleanup and separation protocols, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents;
e) Sample analysis;
f) Standard and reagent origin, receipt, preparation, and use;
g) Equipment receipt, use, specification, operating conditions and preventative maintenance;
h) Calibration criteria, frequency and acceptance criteria;
i) Data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions;
j) Method performance criteria including expected quality control requirements;
k) Quality control protocols and assessment;
l) Electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries;
m) All automated sample handling systems;
n) Records storage and retention; and
o) Sample disposal including the date of sample or subsample disposal and name of the responsible person.

5.12.3.2 Laboratory Support Activities

In addition to documenting all the above-mentioned activities, the following shall be retained:

a) All original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts work sheets and data output records (chromatograms, strip charts, and other instrument response readout records);
b) A written description or reference to the specific method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value;
c) Copies of final reports;
d) Archived standard operating procedures;
e) Correspondence relating to laboratory activities for a specific project;
f) All corrective action reports, audits and audit responses;
g) Proficiency test results and raw data; and
h) Data review and cross checking.

5.12.3.3 Analytical Records

The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, shall include:

a) Laboratory sample ID code;
b) Date of analysis;
c) Instrumentation identification and instrument operating conditions/parameters (or reference to such data);
d) Analysis type;
e) All calculations (automated and manual); and
f) Analyst's or operator's initials/signature.

5.12.3.4 Administrative Records

The following shall be maintained:

a) Personnel qualifications, experience and training records;
b) Initial and continuing demonstration of proficiency for each analyst; and
c) A log of names, initials and signatures for all individuals who are responsible for signing or initialing any laboratory record.

5.12.4 Legal or Evidentiary Custody Procedures

The use of legal chain of custody (COC) protocols is strongly recommended and may be required by some state or federal programs. In addition to the records listed in 5.12.3 and the performance standards outlined in 5.12.1 and 5.12.2, the following protocols shall be incorporated if legal COC is implemented by the organization.

5.12.4.1 Basic Requirements

The chain of custody records shall establish an intact, contiguous record of the physical possession, storage and disposal of sample containers, collected samples, sample aliquots, and sample extracts or digestates. For ease of discussion, the above-mentioned items shall be referred to as samples:

a) A sample is in someone*s custody if:

   1) It is in one*s actual physical possession;

   2) It is in one*s view, after being in one*s physical possession;

   3) It is in one*s physical possession and then locked up so that no one can tamper with it;

   4) It is kept in a secured area, restricted to authorized personnel only.

b) The COC records shall account for all time periods associated with the samples.

c) The COC records shall include signatures of all individuals who had access to individual samples.

d) In order to simplify record-keeping, the number of people who physically handle the sample should be minimized. A designated sample custodian, who is responsible for receiving, storing and distributing samples is recommended.

e) The COC records are not limited to a single form or document. However, organizations should attempt to limit the number of documents that would be required to establish COC.

f) Legal chain of custody shall begin at the point established by the federal or state oversight program. This may begin at the point that cleaned sample containers are provided by the laboratory or the time sample collection occurs.

g) The COC forms shall remain with the samples during transport or shipment.

h) If samples are shipped, the shipping container shall be sealed in such a manner so that tampering by unauthorized personnel is immediately evident.

i) Mailed packages should be registered with return receipt requested. If packages are sent by common carrier, receipts should be retained as part of the permanent chain-of-custody documentation.

j) If required, individual sample containers shall be sealed in such a way to prevent tampering.

k) Once received by the laboratory, laboratory personnel are responsible for the care and custody of the sample and must be prepared to testify that the sample was in their possession and view or secured in the laboratory at all times from the moment it was received from the custodian until the time that the analyses are completed or the sample is disposed.

5.12.4.2 Required Information in Custody Records

In addition to the information specified in 5.11.1.a and 5.11.1.b, tracking records shall include, by direct entry or linkage to other records:

a) Time of day and calendar date of each transfer or handling procedure;

b) Signatures of all personnel who physically handle the sample(s);

c) All information necessary to produce unequivocal, accurate records that document the laboratory activities associated with sample receipt, preparation, analysis and reporting; and

d) Common carrier documents.

5.12.4.3 Controlled Access to Samples

Access to all legal samples and subsamples shall be controlled and documented.

a) A clean, dry, isolated room, building, and/or refrigerated space that can be securely locked from the outside must be designated as a custody room.

b) Where possible, distribution of samples to the analyst performing the analysis must be made by the custodian(s).

c) The laboratory area must be maintained as a secured area, restricted to authorized personnel only.

d) Once the sample analyses are completed, the unused portion of the sample, together with all identifying labels, must be returned to the custodian. The returned tagged sample must be retained in the custody room until permission to destroy the sample is received by the custodian or other authority.

5.12.4.4 Transfer of Samples to Another Party

Transfer of samples, subsamples, digestates or extracts to another party are subject to all of the requirements for legal chain of custody.

5.12.4.5 Sample Disposal

a) If the sample is part of litigation, disposal of the physical sample shall occur only with the concurrence of the affected legal authority, sample data user and/or submitter of the sample.

b) All conditions of disposal and all correspondence between all parties concerning the final disposition of the physical sample shall be recorded and retained.

c) Records shall indicate the date of disposal, the nature of disposal (such as sample depleted, sample disposed in hazardous waste facility, or sample returned to client), and the name of the individual who performed the task.

5.13 LABORATORY REPORT FORMAT AND CONTENTS

The results of each ~~calibration,~~ test, or series of ~~calibrations or~~ tests carried out by the laboratory shall be reported accurately, clearly, unambiguously and objectively, in accordance with any instructions in the ~~calibration or~~ test methods. The results shall **~~should~~ normally be reported in a ~~calibration certificate,~~ test report or ~~test certificate~~ and** shall **~~should~~ include all the information necessary for the interpretation of the ~~calibration or~~ test results and all information required by the method used.**

a) **Each ~~certificate or~~ report shall include at least the following information** (those prefaced with "where relevant" are not mandatory):

1) **a title, e.g. "~~Calibration Certificate~~", "Test Report", or "Test Certificate",** "Certificate of Results" or "Laboratory Results";

2) **name and address of laboratory, and location where the ~~calibration or~~ test was carried out if different from the address of the laboratory** and phone number with name of contact person for questions;

3) **unique identification of the certificate or report (such as serial number) and of each page, and the total number of pages;**
This requirement may be presented in several ways:

i. The total number of pages may be listed on the first page of the report as long as the subsequent pages are identified by the unique report identification and consecutive numbers, or

ii. Each page is identified with the unique report identification, the pages are identified as a number of the total report pages (example: 3 of 10, or 1 of 20).

Other methods of identifying the pages in the report may be acceptable as long as it is clear to the reader that discrete pages are associated with a specific report, and that the report contains a specified number of pages.

4) **name and address of client, where appropriate** and project name if applicable;

5) **description and unambiguous identification of the ~~item calibrated or~~ tested <u>sample</u>** including the client identification code;

6) **characterization and condition of the <u>sample</u> ~~calibration or test item~~;**

7) **date of receipt of sample ~~calibration or test item~~,** date and time of sample collection, **~~and~~ date(s) of performance ~~of calibration or~~ test, ~~where appropriate~~**, and time of sample preparation and/or analysis if the required holding time for either activity is less than or equal to 48 hours;

8) **identification of the ~~calibration or~~ test method used, or unambiguous description of any non-standard method used;**

9) where relevant, **reference to sampling procedure~~, where relevant~~;**

10) **any deviations from, additions to or exclusions from the ~~calibration or~~ test method, and any other information relevant to a specific ~~calibration or~~ test, such as environmental conditions** including the use of relevant data qualifiers;

11) **measurements, examinations and derived results, supported by tables, graphs, sketches and photographs as appropriate, and any failures identified;** A description of the transformations, calculations, or operations performed on the data, a summary and analysis of the data. Where applicable, identification of whether data is calculated on a dry weight or wet weight basis; identification of the reporting units such as ug/l or mg/kg and for Whole Effluent Toxicity, identification of the statistical package used to provide data.

12) where relevant**, a statement of the estimated uncertainty of the ~~calibration or~~ test result ~~(where relevant)~~;**

In situations where required by the client or regulatory agency, this information shall be provided. It may be required of laboratories involved in trace analyses, where there is an uncertainty associated with detection limits.

13) **a signature and title, or an equivalent identification of the person(s) accepting responsibility for the content of the certificate or report (however produced), and date of issue;**

14) **where relevant, a statement to the effect that the results relate only to the items ~~calibrated or~~ tested** or to the sample as received by the laboratory**;**

15) **a statement that the certificate or report shall not be reproduced except in full, without the written approval of the laboratory;**

16) Clear identification of all data provided by outside sources, such as air temperature or ambient water temperature;

b) Laboratories who are operated by a facility and whose sole function is to provide data to the facility management for compliance purposes must provide items 1,3,4,5,7,8,10, and 11 from the above list to management. The facility management must assure that the remaining items are added in the report to the regulatory authority.

c) **Where the certificate or report contains results of ~~calibrations or~~ tests performed by sub-contractors, these results shall be clearly identified.**

d) **Particular care and attention shall be paid to the arrangement of the certificate or report, especially with regard to presentation of the ~~calibration or~~ test data and ease of assimilation by the reader. The format shall be carefully and specifically designed for each type of ~~calibration~~ or test carried out, but the headings shall be standardized as far as possible.**

e) After issuance of the report, the laboratory report shall remain unchanged. **Material amendments to a calibration certificate, test report or test certificate after issue shall be made only in the form of a further document, or data transfer including the statement "Supplement to ~~Calibration Certificate [or~~ Test Report or Test Certificate~~]~~, serial number . . . [or as otherwise identified]", or equivalent form of wording. Such amendments shall meet all the relevant requirements of this** Standard **~~Guide~~.**

f) **The laboratory shall notify clients promptly, in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any calibration certificate, test report or test certificate or amendment to a report or certificate.**

g) **The laboratory shall ensure that, where clients require transmission of ~~calibration or~~ test results by telephone, telex, facsimile or other electronic or electromagnetic means, staff will follow documented procedures that ensure that the requirements of this Standard ~~Guide~~ are met and that confidentiality is preserved.**

h) The laboratory shall certify that the test results meet all requirements of NELAP or provide reasons and/or justification if they do not.

5.14 SUBCONTRACTING ANALYTICAL SAMPLES

a) **Where a laboratory sub-contracts any part of the ~~calibration or~~ testing, this work shall be placed with a laboratory complying with these requirements** (that is the laboratory shall be accredited under NELAC)**. The laboratory shall ensure and be able to demonstrate that its sub-contractor is competent to perform the activities in question and complies with the same criteria of competence as the laboratory in respect to the work being sub-contracted. The laboratory shall advise the client in writing of its intention to sub-contract any portion of the testing to another party.**

b) **The laboratory shall record and retain details of its investigation of the competence and compliance of its sub-contractors and maintain a register of all sub-contracting.** This register shall include all records pertaining to the qualifications of subcontracted laboratories including records of any applicable certifications.

5.15 OUTSIDE SUPPORT SERVICES AND SUPPLIES

a) **Where the laboratory procures outside services and supplies, other than those referred to in this Standard ~~Guide~~, in support of ~~calibrations or~~ tests, the laboratory shall use only those outside support services and supplies that are of adequate quality to sustain confidence in the laboratory's ~~calibrations or~~ tests.**

b) **Where no independent assurance of the quality of outside support services or supplies is available, the laboratory shall have procedures to ensure that purchased equipment, materials and services comply with specified requirements. The laboratory should, wherever possible, ensure that purchased equipment and consumable materials are not used until they have been inspected, calibrated or otherwise verified as complying with any standard specifications relevant to the calibrations or tests concerned.**

c) **The laboratory shall maintain records of all suppliers from whom it obtains support services or supplies required for calibrations or tests.**

5.16 COMPLAINTS

The laboratory shall have documented policy and procedures for the resolution of complaints received from clients or other parties about the laboratory's activities. A record shall be maintained of all complaints and of the actions taken by the laboratory.

Where a complaint, or any other circumstance, raises doubt concerning the laboratory's compliance with the laboratory's policies or procedures, or with the requirements of this Standard Guide or otherwise concerning the quality of the laboratory's calibrations or tests, the laboratory shall ensure that those areas of activity and responsibility involved are promptly audited in accordance with ~~the~~ Section 5.5.3.1.

APPENDIX A
TECHNICAL REFERENCES

40 CFR Part 136, Appendix A, paragraphs 8.1.1 and 8.2

American Association for Laboratory Accreditation 1996. General Requirements for Accreditation

“American National Standards Specification and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs (ANSI/ASQC E-4)”, 1994

Catalog of Bacteria, American Type Culture Collection, Rockville, MD

EPA 2185 - Good Automated Laboratory Practices, 1995

“Glossary of Quality Assurance Terms and Acronyms”, Quality Assurance Division, Office of Research and Development, USEPA

"Guidance on the Evaluation of Safe Drinking Water Act Compliance Monitoring Results from Performance Based Methods", September 30, 1994, Second draft.

International vocabulary of basic and general terms in metrology (VIM): 1984. Issued by BIPM. IEC. ISO. and OIML

ISO 3534-1: “Statistics, vocabulary and symbols - Part 1: Probability and general statistical terms”

ISO 7218: Microbiology - General Guidance for Microbiological Examinations

ISO 8402: 1986. Quality - Vocabulary

ISO 9000: 1987. Quality management and quality assurance standards - Guidelines for selection and use

ISO 9001: 1987. Quality Systems - Model for quality assurance in design/development, production, installation and servicing

ISO 9002: 1987. Quality systems - Model for quality assurance in production and installation

ISO/IEC Guide 2: 1986. General terms and their definitions concerning standardization and related activities

ISO/IEC25: 1990. General requirements for the competence of calibration and testing laboratories

“Laboratory Biosafety Manual”, World Health Organization, Geneva, 1983

Manual for the Certification of Laboratories Analyzing Drinking Water EPA/570/9-90/008

Manual of Method for General Bacteriology, Philipp Gerhard et al., American Society for Microbiology, Washington, 1981

Performance Based Measurement System, EPA EMMC Method Panel, PBM workgroup, 1996

APPENDIX B
DEFINITIONS FOR QUALITY SYSTEMS

The following definitions are used in the text of Quality Systems. In writing this document, the following hierarchy of definition references were used: ISO 8402, ANSI/ASQC E-4, EPA's Quality Assurance Division Glossary of Terms, and finally definitions developed by NELAC and/or the Quality Assurance Standing Committee. The source of each definition is noted.

Acceptable Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accreditation: the process by which an agency or organization evaluates and recognizes a program of study or an institution as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one. (NELAP)

Accrediting Authority: the agency having responsibility and accountability for environmental laboratory accreditation and who grants accreditation. For the purposes of NELAC, this is EPA, other federal agencies, or the state. (NELAP)

Accrediting Body: the organization that actually executes the accreditation process, i.e., receives and reviews accreditation applications, reviews QA documents, reviews proficiency testing results, surveys the site, etc., whether EPA, the state, or contracted private party. (NELAP)

Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Analytical Reagent (AR) Grade: designation for the high purity of certain chemical reagents and solvents given the American Chemical Society. (Quality Systems)

Batch: environmental samples which are prepared and/or analyzed together with the same process and personnel, using

the same lot(s) of reagents, with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. The size of a batch can range from one environmental sample to 20 environmental samples. All environmental samples in the batch must be of the same matrix as defined by NELAC. The resulting extracts, digestates or concentrates may be combined to into any analytical batch. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples. (Quality Systems)

Blank: A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usually analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC, Definitions of Environmental Quality Assurance Terms, 1996)

Blind Sample: a subsample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.

Calibration: the set of operations which establish, under specified conditions, the relationship between values indicated by a measuring instrument or measuring system, or values represented by a material measure, and the corresponding known values of a measurand. (VIM - 6.13)

Calibrate: to determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter or other devise, or the correct value for each setting of a control knob. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements.

Calibration Curve: the graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response.

Calibration Method: Defined technical procedure for performing a calibration.

Calibration Standard: a solution prepared from the primary

dilution standard solution or stock standard solutions and the internal standards and surrogate analytes. The Calibration solutions are used to calibrate the instrument response with respect to analyte concentration. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Certified Reference Material (CRM): A reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body. (ISO Guide 30 - 2.2)

Chain of Custody: an unbroken trail of accountability that insures the physical security of samples, data and records.

Compromised Samples: those samples which were improperly sampled, or with insufficient documentation (chain of custody and other sample records and/or labels), improper preservation and/or containers were used, or the holding time has been exceeded. Under normal conditions compromised samples are not analyzed. If emergency situations require analysis, the results must be appropriately qualified.

Confirmation: verification of the presence of a component through the use of an analytical technique based on a different scientific principle from the original method. These may include:

Second column confirmation
Alternate wavelength
Derivatization
Mass spectral interpretation
Alternative detectors or
Additional cleanup procedures.

Corrective Action: action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Data Audit: a qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality (i.e., that they meet specified acceptance criteria.

Data Reduction: the process of transforming raw data by arithmetic or statistical calculations, standard curves,

concentration factors, etc., and collation into a more useful form.

Document Control: the act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed. (ASQC, Definitions of Environmental Quality Assurance Terms, 1996)

Double Blind Sample: a sample submitted to evaluate performance with concentration and identity unknown to the analyst.

Duplicate Analyses: the analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory.

Holding Times (Maximum Allowable Holding Times): the maximum times that samples may be held prior to analysis and still be considered valid. (40 CFR Part 136).

Initial Demonstration of Analytical Capability: procedure to establish the ability to generate acceptable accuracy and precision which is included in many of the EPA's analytical methods. In general the procedure includes the addition of a specified concentration of each analyte (using a QC check sample) in each of four separate aliquots of laboratory pure water. These are carried through the entire analytical procedure and the percentage recovery and the standard deviation are determined and compared to specified limits. (40 CFR Part 136).

Instrument Blank: a clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Internal Standard: a known amount of standard added to a test portion of a sample and carried through the entire measurement process as a reference for evaluating and controlling the precision and bias of the applied analytical method.

Laboratory: Body that calibrates and/or tests.

NOTES:

1. In cases where a laboratory forms part of an organization that carries out other activities besides calibration and testing, the term "laboratory" refers only to those parts of that organization that are involved in the calibration and testing process.

2. As used herein, the term "laboratory" refers to a body that carries out calibration or testing
   - at or from a permanent location,
   - at or from a temporary facility, or
   - in or from a mobile facility. (ISO ???)

Laboratory Control Sample (quality control sample): an uncontaminated sample matrix spiked with known amounts of analytes from a source independent of the calibration standards. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Laboratory Duplicate: Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.

Legal Chain of Custody (COC): an unbroken trail of accountability that ensures the physical security of samples, data and records. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Manager (however named): the individual designated as being responsible for the overall operation, all personnel, and the physical plant of the environmental laboratory. A supervisor may report to the manager. In some cases, the supervisor and the manager may be the same individual.

Matrix: The component or substrate which contains the analyte of interest. For purposes of batch determination, the following matrix types shall be used:

Drinking water: Any aqueous sample that has been designated a potable or potential potable water source.

Aqueous: Any aqueous sample excluded from the definition of a water matrix or Saline/Estuarine source. Includes surface water, groundwater and effluents.

Saline/Estuarine: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Non-aqueous liquid: Any organic liquid with <15% settleable solids.

Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Solids: Includes soils, sediments, sludges and other matrices with >15% settleable solids.

Chemical Waste: A product or by-product of a industrial process that results in a matrix not previously defined.

Air Samples: Media used to retain the analyte of interest from an air sample such as sorbent tubes or summa canisters. Each medium shall be considered as a distinct matrix. (Quality Systems)

Matrix Spike (spiked sample, fortified sample): prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Matrix Spike Duplicate (spiked sample/fortified sample duplicate): a second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

May: permitted, but not required (TRADE)

Method Blank: a clean sample processed simultaneously with and under the same conditions as samples containing an analyte of interest through all steps of the analytical procedures. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Method Detection Limit (Analytical Detection Limit): the minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136 Appendix B).

Must: denotes a requirement that must be met. (Random House College Dictionary)

Negative Control: measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.

NELAC: National Environmental Laboratory Accreditation Conference. A voluntary organization of state and federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories. A subset of NELAP. (NELAC)

NELAP: the overall National Environmental Laboratory Accreditation Program of which NELAC is a part. (NELAC)

Performance Audit: the routine comparison of independently obtained quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory.

Performance Based Measurement System (PBMS): a set of processes wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate methods to meet those needs in a cost-effective manner.

Proficiency Testing Program: the aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results in comparison to peer laboratories and the collective demographics and results summary of all participating laboratories.

Proficiency Test Sample (PE): a sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified performance limits. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Positive Control: measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.

Precision: the degree to which a set of observations or measurements of the same property, usually obtained under

similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Preservation: refrigeration and or reagents added at the time of sample collection to maintain the chemical and or biological integrity of the sample.

Proficiency Testing: Determination of the laboratory calibration or testing performance by means of interlaboratory comparisons. (ISO/IEC Guide 2 - 12.6, amended)

Protocol: a detailed written procedure for field and/or laboratory operation (e.g., sampling, analysis) which must be strictly followed.

Pure Reagent Water: shall be ASTM Type I or Type II water in which no target analytes or interferences are detected as required by the analytical method.

Quality Assurance: an integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Quality Control: the overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Quality Control Sample: an uncontaminated sample matrix spiked with known amounts of analytes from a source independent from the calibration standards. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Quality Manual: A document stating the quality policy, quality system and quality practices of an organization. This may be also called a Quality Assurance Plan or a Quality Plan.

NOTE - The quality manual may call up other documentation relating to the laboratory's quality arrangements.

Quality System: a structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC. (ANSI/ASQC E-41994)

Range: the difference between the minimum and the maximum of a set of values.

Raw Data: any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes ,or exact copies thereof and that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted.

Reagent Blank (method reagent blank): a sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Reference Material: A material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30 - 2.1)

Reference Standard: A standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM - 6.08)

Requirement: A translation of the needs into a set of individual quantified or descriptive specifications for the characteristics of an entity in order to enable its realization and examination.

Reference Toxicant: see D.2.1.a

Replicate Analyses: the measurements of the variable of interest performed identically on two or more subsamples of the same sample within a short time interval.

Sample Duplicate: two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method including sampling and analysis. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Selectivity: (Analytical chemistry) the capability of a method or instrument to respond to a target substance or constituent in the presence of nontarget substances.

Sensitivity: the capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.

Shall: denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there be no deviation. This does not prohibit the use of alternative approaches or methods for implementing the specification so long as the requirement is fulfilled. (*Style Manual for Preparation of Proposed American National Standards*, American National Standards Institute, eighth edition, March 1991).

Should: denotes a guideline or recommendation whenever noncompliance with the specification is permissible. (*Style Manual for Preparation of Proposed American National Standards*, American National Standards Institute, eighth edition, March 1991).

Standard Operating Procedures (SOPs): a written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Spike: a known mass of target analyte added to a blank sample or subsample; used to determine recovery efficiency or

for other quality control purposes.

Standard Reference Material (SRM): a certified reference material produced by the U.S. National Institute of Standards and Technology and characterized for absolute content, independent of analytical method.

Supervisor (however named): the individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses.

Surrogate: a substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Systems Audit (also Technical Systems Audit): a thorough, systematic on-site, qualitative review of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system.

Technical Director: *Definition needs to be developed*

Technical Employee: the designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent Quality Controls to meet the required level of quality.

Test: A technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process or service according to a specified procedure.

NOTE - The result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2 - 12.1, amended)

Test Method: Defined technical procedure for performing a

test.

Testing Laboratory: Laboratory that performs tests. (ISO/IEC Guide 2 - 12.4)

Traceability: The property of a result of a measurement whereby it can be related to appropriate standards, generally international or

national standards, through an unbroken chain of comparisons. (VIM - 6.12)

Test Sensivity/Power: D.2.4.a

Verification: Confirmation by examination and provision of evidence that specified requirements have been met.

NOTE - In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment.

The result of verification leads to a decision either to restore in service, to perform adjustments, or to repair, or to downgrade, or to declare obsolete. In all cases it is required that a written trace of the verification performed shall be kept on the measuring instrument's individual record.

Validation: the process of substantiating specified performance criteria.

Verification: the process of establishing or confirming the truth or factualness of data.

APPENDIX C
CHECKLIST FOR DEMONSTRATION OF METHOD PERFORMANCE

PROCEDURE FOR INITIAL DEMONSTRATION OF CAPABILITY

An initial demonstration of method performance must be made prior to using any method, and at any time there is a change of equipment personnel or procedure (see 5.10.2.1).

All initial demonstrations, continuing demonstrations and method certification shall be documented through the use of the forms in this appendix.

The following steps, which are adapted from the EPA methods published in 40 CFR Part 136, Appendix A, shall be performed:

a) A quality control check sample concentrate shall be obtained from EPA or a certified source. If not available, the QC check sample may be prepared by the laboratory using stock standards that are prepared independently from those used in instrument calibration.

b) The concentrate shall be diluted in a volume of laboratory pure water sufficient to prepare four aliquots at the required method volume to a concentration approximately 10 times the method-stated or laboratory-calculated method detection limit.

c) The four aliquots shall be prepared and analyzed according to the method.

d) Using the four results, calculate the average recovery ($\bar{x}$) in the appropriate reporting units (such as μg/L) and the standard deviation (s) (in the same units) for each parameter of interest.

e) For each parameter, compare s and $\bar{x}$ to the corresponding acceptance criteria for precision and accuracy in the method (if applicable) or laboratory-generated acceptance criteria (if a non-standard method). If s and $\bar{x}$ for all parameters meet the acceptance criteria, the analysis of actual samples may begin. If any one of the parameters exceed the acceptance range, the performance is unacceptable for that parameter.

f) When one ore more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed

according to 1) or 2) below.

1) Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with c) above.

2) Beginning with c) above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with c).

Method Performance System Certification Statement

Date: **Page __of __**

Facility Name:

Discharge Point ID (where applicable):

EPA Program and Applicable Regulation:

Medium:
(i.e., wastewater, drinking water, soil, air, waste solid, leachate, sludge, other)

Analyte, Class of Analytes or Measureand (CAS # where available)
(i.e , barium, trace metals, benzene, volatile organics, etc.)

We, the undersigned, CERTIFY that:

1. the methods in use at this facility for the analyses of samples for the Programs of the U.S. Environmental Protection Agency, have met the Initial and any required Continuing Demonstration of Method Performance Criteria specified under the Performance-Based System.

2. A copy of the Performance-Based Method, written in EMMC format, and copies of the reference method and laboratory-specific SOPs are available for all personnel on-site.

3. The data and checklists associated with the initial and continuing demonstration of method performance are true, accurate, complete and self-explanatory[1].

4. All raw data (including a copy of this certification

True: Consistent with supporting data.

Accurate: Based on good laboratory practices consistent with sound scientific principles/practices.

Complete: Includes the results of all supporting performance testing.

Self-Explanatory: Data properly labeled and stored so that the results are clear and require no additional explanation.

form) necessary to reconstruct and validate these performance related analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized inspectors.

Laboratory Manager’s Name and Title Signature Date

Quality Assurance Officer’s Name Signature Date

This certification form must be completed when the performance-based method is originally certified, each time the continuing demonstration of method performance is documented and whenever a change of personnel involves the Laboratory Manger or the Quality Assurance Officer.

Checklist for Initial Demonstration of Method Performance

***Provide a checklist for each matrix included in the demonstration*.**

Date: **Page __of __**

Laboratory ~~Facility~~ Name:

Discharge Point ID (where applicable):

EPA Program and Applicable Regulation:

Medium:
(i.e., wastewater, drinking water, soil, air, waste solid, leachate, sludge, other)

Analyte, Class of Analytes of Other Measureand:
(i.e., barium, trace metals, benzene, volatile organics, etc.)

Initial Demonstration of Method Performance *(1)*				
Category	**Performance Criteria *(2)* Based on**		**Results Obtained**	**Perf. Spec. Achieved (✓)**
	Reference Method	**Measurement Quality Objective**		
1. Written method (addressing all elements in the EMMC format) attached				
2. Title, number and date/rev. of "reference method", if applicable ***(3)***				
3. Copy of the reference method, if applicable, maintained at facility				
4. Differences between PBM and reference method (if applicable) attached				
5. Concentrations of calibration standards				
6. %RSD or slope/correlation coefficient of calibration regression				

Initial Demonstration of Method Performance *(1)*				
Category	**Performance Criteria *(2)* Based on**		**Results Obtained**	**Perf. Spec. Achieved (✓)**
	Reference Method	**Measurement Quality Objective**		
7. Performance range tested (with units)				
8. Sample(s) used in initial demonstration have recommended preservative, where applicable.				
9. Samples(s) used in initial demonstration met recommended holding times, where applicable				
10. Interferences				
11. Qualitative identification criteria used				
12. Performance Evaluation studies performed for analytes of interest, where available: Last study sponsor and title: Last study number:				
13. Analysis of external reference material Last study sponsor and title: Last study number:				
14. Authenticity of reference material				
15. Surrogates used, if applicable				
16. Concentrations of surrogates, if applicable				
17. Recoveries of Surrogates appropriate to the proposed use, if applicable				
18. Sample preparation				
19. Clean-up procedures				

Initial Demonstration of Method Performance *(1)*				
Category	**Performance Criteria *(2)* Based on**		**Results Obtained**	**Perf. Spec. Achieved (✓)**
	Reference Method	**Measurement Quality Objective**		
20. Method Blank				
21. Matrix (reagent water, drinking water, soil, waste solid, air, etc.)				
22. Spiking system, appropriate to method and application				
23. Spike levels (w/ units corresponding to final sample concentration)				
24. Source of spiking material				
25. Number of spikes				
26. Precision (analyte by analyte)				
27. Bias (analyte by analyte)				
28. Detection Limit (w/ units; analyte by analyte)				
29. Confirmation of Detection Limit				
30. Quantiation Limit (w/ units: analyte by analyte)				
31. Frequency (Initial Demonstration to be performed)				
32. Other criterion (specify)				
33. Other criterion (specify)				
34. Other criterion (specify)				

[1] Provide a detailed narrative description of the initial demonstration.

[2] For multi-analyte methods, enter the range of performance criteria and attach an analyte-specific list.

[3] If a reference method is the source of the performance criteria, the reference method should be appropriate to the required application, and the listed criteria should be fully consistent with that reference method.

Name and signature of each analyst involved in the initial demonstration of method performance (includes all steps in the proposed method/modification):

_____	_____	_____
Name	**Signature**	**Date**
_____	_____	_____
Name	**Signature**	**Date**
_____	_____	_____
Name	**Signature**	**Date**

The certification above must accompany this form each time it is submitted.

Checklist for Continuing Demonstration of Method Performance

Date: **Page __of __**

Facility Name:

Discharge Point ID, where applicable:

EPA Program and Applicable Regulation:

Medium:
(i.e., wastewater, drinking water, soil, air, waste solid, leachate, sludge, other)

Analyte, Class of Analytes or Measureand (CAS # where available)
(i.e., barium, trace metals, benzene, volatile organics, etc.)

Continuing Demonstration of Method Performance				
Category	Required Frequency	Specific Performance Criteria	Results Obtained	Perf. Spec. Achieved (✓)
1. Method blank (taken through all steps in the procedure)				
2. Concentrations of calibration standards used to verify working range (with units), where applicable				
3. Calibration verification				
4. Calibration check standard				
5. External QC sample (where available)				
6. Performance evaluation (PE) studies, if applicable Last study sponsor and title: Last study number:				
7. List analytes for which results were "not acceptable" in PE study	----	----	----	----
8. Surrogates used, if applicable				
9. Concentration/s of Surrogates, if applicable				
10. Recovery of Surrogates (acceptance range for multianalyte methods), if applicable				
11. Matrix				
12. Matrix spike compounds				
13. Concentration of Matrix spike compounds				

14. Recoveries of Matrix spike compounds				
15. Qualitative identification criteria used				
16. Sample preparation				
17. Clean-up procedures				
18. Confirmation				
19. Other category (specify)				
20. Other category (specify)				

Name and signature of each analyst involved in continuing demonstration of method performance (includes all steps in the proposed method/modification):

_____ _____ _____

Name **Signature** **Date**

_____ _____ _____

Name **Signature** **Date**

_____ _____ _____

Name **Signature** **Date**

The certification above must accompany this form each time it is submitted.

APPENDIX D

ESSENTIAL QUALITY CONTROL REQUIREMENTS

The method specified and/or method-recommended quality control protocols shall be followed. The essential standards outlined in this appendix shall be used if no protocols are written into the method or if the method protocols are less stringent.

D.1 Chemical Testing

D.1.1 Positive and Negative Controls

a) Negative Controls

1) Method Blanks - Shall be performed at a frequency of one per batch of samples per matrix type per sample extraction or preparation method. The results of this analysis shall be one of the QC measures to be used to assess batch acceptance. If blank contamination is found, the analysis of all samples associated with the blank must be stopped until the source of the contamination is identified and measures are taken to correct, minimize or eliminate the problem. The results of samples affected by the contaminated blank shall either be reprocessed for analysis or reported with appropriate data qualifying codes.

b) Positive Controls

1. Matrix Spikes (MS) - Shall be performed at a frequency of one in 20 samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as, total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a matrix spike may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the spike.

2) Laboratory Control Sample - (QC Check Samples) Shall be analyzed at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as total suspended solids, total dissolved

solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples shall be used to determine batch acceptance. NOTE: the Matrix spike (see 1 above) may be used as a control as long as the acceptance criteria are as stringent at the LCS.

3) Surrogates - Surrogate compounds must be added to all samples, standards, and blanks, whenever possible, for all organic chromatography methods. The acceptance criteria specified by the method shall be used to evaluate sample acceptance.

4) The laboratory is expected to spike all method-listed constituents. However, in cases where the constituents interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the method has an extremely long list of constituents (such as Method 8270 or 6010) or constituents are incompatible, a representative number (10%) of the listed components may be used to control the method. The selected components shall represent all chemistries, elution patterns and masses and shall include permit specified analytes and other client requested components. The laboratory shall ensure, however, that all method listed components are used in the spike mixtures within a two-year time period, and that no one component or components dominate the spike mixture.

D.1.2 Analytical Variability/Reproducibility

Matrix Spike Duplicates (MSDs) or Laboratory Duplicates -Shall be analyzed at a minimum of 1 in 20 samples per matrix type per sample extraction or preparation method. The laboratory shall document their procedure to select the use of appropriate type of duplicate. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in the duplicates may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the duplicate.

D.1.3 Method Evaluation

In order to ensure the accuracy of the reported result, the following procedures shall be in place:

a) Initial Demonstration of Analytical Capability (Section 5.10.2.1) shall be performed initially and with a significant

change, such as a new analyst, instrument or technique.

b) Calibration - Calibration protocols specified in Section 5.10.1 shall be followed.

c) Proficiency Test Samples - The results of such analyses (5.4.2.j or 5.5.3.4) shall be used by the laboratory to evaluate the ability of the laboratory to produce accurate data.

D.1.4 Sensitivity

a) Method detection Limits - Method detection limits shall be determined by 40 CFR Part 136, Appendix B unless specified by a method or program. The detection limit shall be initially determined for the compounds of interest in each method in laboratory pure water or the matrix of interest and shall be verified annually. All procedures used must be documented including the matrix type.

D.1.5 Data Reduction

The procedures for data reduction, such as use of linear regression, shall be documented.

D.1.6 Quality of Standards and Reagents

a) The source of standards shall comply with 5.9.2.

b) Reagent Quality, Water Quality and Checks:

1) Reagents - In methods where the purity of reagents is not specified, analytical reagent grade shall be used. Reagents of lesser purity than those specified by the method shall not be used. The labels on the container should be checked to verify that the purity of the reagents meets the requirements of the particular method. Such information shall be documented.

2) Water - The quality of water sources shall be monitored and documented and shall meet method specified requirements.

D.1.7 Selectivity

a) Absolute retention time and relative retention time aid in the identification of components in chromatographic analyses and to evaluate the effectiveness of a column to separate constituents. The laboratory shall develop and document acceptance criteria for retention time windows.

b) A confirmation shall be performed to verify the compound

identification when positive results are detected on an uncharacterized samples. Such confirmations shall be performed on organic tests such as pesticides, herbicides, or acid extractable or when recommended by the analytical method except when the analysis involves the use of a mass spectrometer. Confirmation is required unless stipulated in writing by the client. All confirmation shall be documented.

D.1.8 Constant and Consistent Test Conditions

a) The laboratory shall assure that the test instruments consistently operate within the specifications of the test methods and equipment manufacturer.

b) Glassware Cleaning - Glassware shall be cleaned to meet the sensitivity of method.

Any cleaning and storage procedures that are not specified by the method shall be documented in laboratory records and SOPs.

D.2 Whole Effluent Toxicity

D.2.1 Positive and Negative Controls

a) Positive Control - Reference Toxicants - Reference toxicant tests indicate the sensitivity of the test organisms being used and demonstrate a laboratory's ability to obtain consistent results with the method.

1) The laboratory must demonstrate its ability to obtain consistent, results with reference toxicants before it performs toxicity tests with effluents for permit compliance purposes.

i An intralaboratory coefficient of variation (%CV) is not established for each test method. However, a testing laboratory shall maintain control charts for the control performance and reference toxicant statistical endpoint (such as NOEC or ECp) and shall evaluate the intralaboratory variability with a specific reference toxicant for each method. In addition, a laboratory must produce test results that meets test acceptability criteria (such as such as greater than 80% survival in the control) as specified in the specific test method.

ii Intra-laboratory precision on an ongoing basis must be

determined through the use of reference toxicant tests and plotted in quality control charts. As specified in the test methods, the control charts shall be plotted as point estimate values, such as EC25 for chronic tests and LC 50 for acute tests, over time within a laboratory.

2) Frequency:

i The frequency of reference toxicant testing shall comply with the EPA or state permitting authority requirements.

ii Reference toxicant tests shall be performed at least once a month if using test organisms cultured within the testing laboratory during that month.

3) The USEPA test methods for EPA/600/4-91-002, EPA/600/4-91-003 and EPA/600/4-90-027F do not currently specify a particular reference toxicant and dilution series, however, if the state or permitting authority identifies a reference toxicant or dilution series for a particular test, the laboratory shall follow the specified requirements.

4) Test Acceptability Criteria (TAC) - The test acceptability criteria (for example, the chronic *Ceriodaphnia* test, requires 80% or greater survival and an average 15 young per female in the controls) as specified in the test method must be achieved for both the reference toxicant and effluent test. The criteria shall be calculated and shall meet the method specified requirements for performing toxicity:

i The control population of *Ceriodaphnia* shall contain no more than 20% males.

ii An individual test may be conditionally acceptable if temperature, dissolved oxygen, pH and other specified conditions fall outside specifications, depending on the degree of the departure and the objectives of the tests (see test conditions and test acceptability criteria specified for each test method). The acceptability of the test shall depend on the experience and professional judgment of the technical employee and the permitting authority.

b) Negative Control - Control, Brine Control or Dilution Water - The standards for the use, type and frequency of testing are

specified by the methods and by permit and shall be followed.

D.2.2 Variability and/or Reproducibility

Intra-laboratory precision shall be determined on an ongoing basis through the use of further reference toxicant tests and related control charts as described in item 5.5.4.2.1 a) above.

D.2.3 Accuracy

This principle is not applicable to Whole Effluent Toxicity.

D.2.4 Test Sensitivity

a) Test sensitivity (or test power) of the tests will depend in part on the number of replicates per concentration, the significance level selected (0.05), and the type of statistical analysis. If the variability remains constant, the sensitivity of the test will increase as the number of replicates is increased. Test sensitivity is the minimum significant difference (MSD) between the control and test concentration that is statistically significant. If the Dunnett's procedure is used, the MSD shall be calculated according to the formula specified by the EPA method and reported with the test results.

b) For non-normal distribution and or heterogenous variances the MSD can be estimated, but is not required.

c) Point estimates: (LCp, ICp, or Ecp) - Confidence intervals shall be reported as a measure of the precision around the point estimate value.

d) The MSD shall be calculated and reported for only chronic endpoints. In addition, the calculated endpoint is typically a lethal concentration of 50% (LC 50), therefore, confidence intervals shall be reported as a measure of the precision around the point estimate value. In order to have sufficient replicates to perform a reliable MSD, such tests shall have a minimum of four replicates per treatment so that either parametric or non parametric tests can be conducted.

D.2.5 Selection of Appropriate Statistical Analysis Methods

a) The methods of data analysis and endpoints will be specified by language in the permit or, if not present in the permit, by the EPA methods manuals for Whole Effluent Toxicity.

b) Dose Response Curves - When required, the data shall be plotted in the form of a curve relating the dose of the chemical to cumulative percentage of test organisms demonstrating a response such as death.

D.2.6 Selection and Use of Reagents and Standards

a) The grade of all reagents used in Whole Effluent Toxicity tests is specified in the method except the reference standard. All reference standards shall be prepared from

chemicals which are analytical reagent grade or better. The preparation of all standards and reference toxicants shall be documented.

b) All standards and reagents associated with chemical measurements, such as dissolved oxygen, pH or specific conductance, shall comply with the standards outlined in 5.5.4.1 above.

D.2.7 Selectivity

This principle is not applicable. The selectivity of the test is specified by permit.

D.2.8 Constant and Consistent Test Conditions

a) If closed refrigerator-sized incubators are used, culturing and testing of organisms shall be separated to avoid loss of cultures due to cross-contamination.

b) The laboratory or a contracted outside expert shall positively identify test organisms to species on an annual basis. The taxonomic reference (citation and page(s))and the names(s) of the taxonomic expert(s) must be kept on file at the laboratory.

c) Instruments used for routine measurements of chemical and physical parameters such as pH, DO, conductivity, salinity, alkalinity, hardness, chlorine, and weight shall be calibrated, and/or standardized per manufacturer's instructions and Section 5.5.4.1. Temperature shall be calibrated per section 5.9.5.2.3. All measurements and calibrations shall be documented.

d) Test temperature shall be maintained as specified in the methods manuals. The average daily temperature of the test solutions must be maintained within 1°C of the selected test temperature, for the duration of the test. The minimum frequency of measurement shall be once per 24 hour period. The test temperature for continuous flow toxicity tests shall be recorded and monitored continuously.

e) Water used for culturing and testing shall be analyzed for toxic metals and organics annually or whenever the minimum acceptability criteria for control survival, growth or reproduction are not met and no other cause, such as contaminated glassware or poor stock, can be identified. The method specified analytes and concentration levels shall be followed.

f) New batches of food used for culturing and testing shall be

analyzed for toxic organics and metals. If food combinations or recipes are used, analyses shall be performed on the final product upon the use of new lot of any ingredient. If the concentration of total organic chlorine exceeds 0.15 ug/g wet weight, or the total concentration of organochlorine pesticides plus PCBs exceeds 0.30 ug/g wet weight, or toxic metals exceeds 20 ug/g wet weight, the food must not be used.

g) Test chamber size and test solution volume shall be as specified in the methods manuals.

h) Test organisms shall be fed the quantity and type food specified in the methods manuals. They shall also be fed at the intervals specified in the methods.

i) Light intensity shall be maintained as specified in the methods manuals. Measurements shall be made and recorded on a yearly basis. Photoperiod shall be maintained as specified in the methods and shall be documented at least quarterly. For algal tests, the light intensity shall be measured and recorded at the start of each test.

j) At a minimum, during chronic testing DO and pH shall be measured daily in at least one replicate of each concentration. DO may be measured in new solutions prior to organism transfer, in old solutions after organisms transfer, or both.

k) All cultures used for testing shall be maintained as specified in the methods manuals.

l) Age and the age range of the test organisms must be as specified in the manuals.

m) The maximum holding time (lapsed time from sample collection to first use in a test) shall not exceed 36 hours without the permission of the permitting authority.

n) All samples shall be chilled to 4°C during or immediately after collection. They shall be maintained at 0.1 to 6°C and the arrival temperature shall be no greater than 6°C. Samples that are hand delivered to the laboratory immediately after collection (i.e., within 1 hour) may not meet the laboratory temperature acceptance criteria. In these cases, the laboratory may accept the samples if there is evidence (such as arrival on ice) that the chilling process has begun.

o) Organisms obtained from an outside source must be from the same batch.

D.3 Microbiology

These standards apply to laboratories undertaking the examination of materials, products and substances involving microbiological analysis, recovery or testing. The procedures involve the culture media, the test sample and the microbial species being isolated, tested or enumerated.

a) Microbiological testing refers to and includes the detection, isolation, enumeration and identification of microorganisms and their metabolites, as well as sterility testing. It includes assays using microorganisms as part of a detection system and their use for ecological testing.

b) These standards are concerned with the quality of test results and not specifically with health and safety measures. In the performance of microbiological testing, safety and health matters must always be considered and conform with regulatory and national policies in this area.

c) Clothing appropriate to the type of testing being performed should be worn, and often includes protection for hair, beard, hands and shoes. Clothing worn in the microbiological laboratory should be removed before leaving the area.

D.3.1 Positive and Negative Controls

a) Negative Controls

The laboratory shall demonstrate that the cultured samples have not been contaminated through sampling handling/preparation or environmental exposure. These controls shall include sterility checks of media and blanks such as filtration blanks.

1) All blanks and uninoculated controls specified by the method shall be prepared and analyzed at the frequency stated in the method.

2) A minimum of one uninoculated control shall be prepared and analyzed unless the same equipment is used to prepare samples for incubation (such as a filtration unit). In such cases, the laboratory shall prepared a series of blanks using the equipment. At least one

beginning and ending control shall be prepared, with additional controls inserted after every 10 samples.

b) Positive Controls

Positive controls demonstrate that the medium can support the growth of the test organism, and that the medium produces the specified or expected reaction to the test organism,.

On a monthly basis, at least one pure culture of a known positive reaction shall be included with the sample test batch.

D.3.2 Test Variability/Reproducibility

a) Duplicates - At least 5% of the positive samples shall be duplicated. In laboratories with more than one analyst, each shall make parallel analyses on at least one positive sample per month.

b) Where possible, participation in, or organization of collaborative trails, proficiency testing, or interlaboratory comparisons, either formal or informal, must be done.

D.3.3 Method Evaluation

a) In order to demonstrate the suitability of a method for specified purpose, an intended purpose, the laboratory shall establish, through method validation, a set of acceptance criteria for the performance characteristics of the method unless such criteria are specified by the method. These criteria must demonstrate that the method provides a correct/expected result with respect to specified limits of detection, selectivity, repeatability, sensitivity and reproductivity.

   1) Accepted (official) methods or commercialized test kits For official methods, or methods from recognized national or international standard organizations, may not require a full validation. Laboratories are required, however, to demonstrate proficiency with the method prior to first use.

   2) Qualitative microbiological test methods in which the response is expressed in terms of presence/absence, shall be validated by estimating, if possible, the specificity, relative trueness, positive deviation, negative deviation, repeatability, reproducibility and the limit of determination within a defined variability. The differences due to the matrices must be taken into account when testing different sample types.

   3) The validation of microbiological test methods shall be performed under the same conditions as those of a real assay. This can be achieved by using a combination of naturally contaminated products and spiked products.

   4) All validation data shall be recorded and stored at least as long as the method is in force, or if withdrawn

from active use, for at least 5 years past the date of last use.

b) Laboratories shall participate in the proficiency test program specified by NELAC (5.4.2.j or 5.5.3.4). Further, laboratories should regularly participate in schemes which are relevant to their scope of accreditation. Such program provide an independent means by which a laboratory may objectively assess and demonstrate the reliability and trueness of results produced by its analytical methods.

D.3.4 Test Performance

All growth and recovery media must be checked to assure that the target organisms respond in an acceptable and predictable manner (see D.3.b).

D.3.5 Data Reduction

a) The calculations, data reduction and statistical interpretations specified by each method shall be followed.

b) If the method specifies colony counts, such as membrane filter or colony counting, then the ability of individual analysts to count colonies shall be verified at least once per month, by having two or more analysts count colonies from the same plate.

D.3.6 Quality of Standards, Reagents and Media

The laboratory shall ensure that the quality of the reagents and media used is appropriate for the test concerned.

a) Culture media may be prepared in the laboratory from the different chemical ingredients, from commercial dehydrated powders or may be purchased ready to use.

b) Reagents and commercial dehydrated powders shall be consumed within the shelf-life of the product and shall be documented according to 5.9.4. The laboratory shall retain all manufacturer supplied “quality specification statements” which may contain such information as shelf life of the product, storage conditions, sampling regimen/rate, sterility check including acceptability criteria, efficacy checks including the organism used, their culture collection reference and acceptability criteria, date of issue of specification, or statements assuring that the relevant

product batch meets the product specifications.

c) Distilled water, deionized water or reverse osmosis produced water free from bactericidal and inhibitory substances shall be used in the preparation of media solutions and buffers. Where required by the method, the quality of the water (such as pH, chlorine residual, specific conductance or metals) shall be monitored at the specified frequency and evaluated according to the stated standards. Records shall be maintained on all activities.

d) Media, solutions and reagents shall be prepared, used and stored according to a documented procedure following the manufacturer's/author's instructions.

e) All laboratory media shall be checked to ensure they support the growth of specific microbial cultures. In addition, selective media should be checked to ensure they suppress the growth of non-target organisms. In preference to using the commonly used streak method, it is better to use a quantitative procedure, where a know (often low) number of relevant organisms are inoculated into the medium under test and the recovery evaluated.

f) Each lot of laboratory detergent shall be checked to ensure that residues from the detergent do not inhibit or promote growth of microorganisms.

D.3.7 Selectivity

a) All confirmation/verification tests specified by the method shall be performed according to method protocols.

b) In order to demonstrate traceability and selectivity, laboratories shall use reference cultures (See Figure D-1) of microorganisms obtained from a recognized national collection or an organization recognized by the accreditation body.

1) Reference cultures may be subcultured once to provide reference stocks. Appropriate purity and biochemical checks shall be made and documented. The reference stocks shall be preserved by a technique which maintains the desired characteristics of the strains. Examples of such methods are freeze-drying, liquid nitrogen storage and deep-freezing methods. Reference stocks shall be used to prepare working stocks for routine work. If

reference stocks have been thawed, they must not be re-frozen and re-used.

2) Bacterial working stocks shall not be sub-cultured under normal conditions. However working stocks may be subcultured up to a defined number of subcultures when:
   i it is required by standard methods or
   ii laboratories can provide documentary evidence demonstrating that there has been no loss of viability, no changes in biochemical activity and/or no change in morphology.

3) Working stocks shall not be subcultured to replace reference stocks.

4) A scheme for handling reference cultures is included in figure D.1.

D.3.8 Constant and Consistent Test Conditions

a) The laboratory shall devise an appropriate environmental monitoring program to indicate trends in levels of contamination appropriate to the type of testing being carried out. Acceptable background counts shall be determined and there shall be a documented procedures to deal with situations in which these limits are exceeded.

b) Walls, floors, ceilings and work surfaces should be non-absorbent and easy to clean and disinfect. Wooden surfaces of fixtures and fitting shall be adequately sealed. Measures should be taken to avoid accumulation of dust by the provision of sufficient storage space by having minimal paperwork in the laboratory and by prohibiting plants and personal possessions from the laboratory work area.

c) Temperature measurement devices

1) Where the accuracy of temperature measurement has a direct effect on the result of the analysis, temperature measuring devices such as liquid-in-glass thermometers, thermocouple, platinum resistance thermometers used in incubators, autoclaves and other equipment shall be the appropriate quality to achieve the specification in the test method. The graduation of the temperature measuring devices must be appropriate for the required accuracy of measurement and they shall be calibrated to national or international standards for temperature (see 5.9.5.2.3).

2) The stability of temperature, uniformity of temperature distribution and time required to achieve equilibrium conditions in incubators, waterbaths, ovens and temperature controlled rooms shall be established, for example, position, space between and height of stacks of Petri dishes.

d) Autoclaves

1) The performance of each autoclave shall be initially

evaluated by establishing its functional properties, for example heat distribution characteristics with respect to typical uses. Autoclaves shall be capable of meeting specified temperature tolerances. Pressure cookers fitted only with a pressure gauge are not recommended for sterilization of media or decontamination of wastes.

2) Records of autoclave operations including temperature and time shall be maintained. This shall be done for every cycle. Acceptance/rejection criteria shall be established and used to evaluate the autoclave efficiency and effectiveness.

e) Volumetric equipment such as automatic dispensers, dispenser/diluters, mechanical hand pipettes and disposal pipettes may all be used in the microbiology laboratory. Regular checks as outlined in Section 5.9.5.2.4 shall be performed and documented.

f) Conductivity meters, oxygen meters, pH meters, hygrometers, and other similar measurement instruments shall be calibrated according to the method specified requirements (see 5.9.5). Timers shall be checked regularly to ensure accurate timing.

D.4 Radioanalysis

A radioanalytical laboratory shall analyze both intralaboratory and interlaboratory quality control samples on a routine basis as described in the laboratory's Quality Assurance Plan. These quality control samples shall account for at least l0 percent of the total analytical load and shall not be limited to one type or category. Where possible, the laboratory shall employ the use of blind and double-blind quality control samples.

D.4.1 Positive and Negative Controls

a) Negative Control - Reagent Blank - The laboratory shall analyze reagent blanks so as to provide a means of evaluating and quantifying potential contamination resulting from the samples' passage through the analytical process. The volume or weight of the blank shall be approximately equal to the volume or weight of samples routinely analyzed, and the blanks shall be carried through the entire analytical process. These blanks shall be included with each batch of samples that must be prepared (that is, chemically separated) prior to analysis.

b) Positive Control - Matrix Spike - The laboratory shall analyze matrix spike samples to evaluate the effect of the sample matrix upon the analytical methodology. The activity level of the matrix spike sample shall be comparable to the activity levels of samples routinely analyzed. The laboratory shall prepare and analyze matrix spike samples for each type of matrix and method. Matrix spike samples shall be performed initially for each matrix type, and verified annually. If a sample matrix is determined to be substantially different from the original matrix (for example, the original was pristine ground water, and the submitted samples are effluent from a wastewater treatment facility) the laboratory shall establish and characterize the "new matrix" with spike samples.

D.4.2 Laboratory Variability/Reproducibility

The laboratory shall perform replicate analyses to evaluate the precision of an analysis. The size of replicate samples shall be approximately equal to the size of samples routinely analyzed.

a) Replicates of actual samples shall be analyzed at least once per quarter.

b) Replicates of matrix spikes samples shall be analyzed at the same frequency as matrix spikes (D.4.1.b).

c) Replicates of traceable reference material (D.4.3.a) shall be analyzed at least annually.

D.4.3 Method Evaluation

a) Traceable Reference Material - The laboratory shall analyze traceable reference materials to evaluate the accuracy and precision of an analytical methodology or an analyst. Traceable Reference Material, as defined by ANSI N42.2 *Measurement Quality Assurance For Radioassay Laboratories* is a NIST prepared standard reference material (SRM) or a sample of known concentration prepared from a NIST traceable reference material (derived standard material). The material shall be analyzed initially, and on a continuing annual frequency.

b) Calibration of Radiation Measurement Systems - All measures shall be performed on an annual basis, and at any time there is an equipment change or whenever significant changes in the analytical instrumentation is detected.

1) Performance assessments shall, at a minimum, utilize the following practices: (l) baselining each measurement system's response characteristics, (2) stability check with control chart plot, and (3) background check with control chart plot. The frequency of the performance assessments must be sufficient to promptly identify and correct invalid results and must also consider radiation measurement system (RMS). For a RMS which is used to identify radionuclides, the stability check shall also include geometry-specific energy-calibration sources.

2) Radiation instrumentation energy calibration (channel number of the multichannel analyzer versus the radiation energy), full-energy peak efficiency, radionuclide activity (concentration) precision and test method precision and bias should be checked with appropriate standard sources.

c) Quality Control Assessment - All measures shall be trended by statistical techniques to establish acceptance criteria. The laboratory shall establish and follow a written standard operating procedure that outlines the protocols to be followed should any quality control measure exceed the established acceptance criteria.

D.4.4 Sensitivity

a) Counter Background - A major factor in determining the sensitivity of measurement procedures is the background of the counter. Therefore, the counter background must be reduced as much as practicable.

1) Where applicable, anticoincidence counting methods shall be used to reduce the influence of cosmic ray cascade radiation interferences.

2) The background of the counter must be minimized by preventing counter contamination with radioactive materials.

3) Backgrounds shall be recorded on each day’s use. These records shall be statistically analyzed to determine correction factors and acceptance ranges.

b) The Environmental Detection Limit (EDL) (that is, the smallest level at which a radionuclide in an environmental medium can be unambiguously distinguished for a given

confidence interval using a particular combination of sampling and measurement procedures, sample size, analytical detection limit, and processing procedure) shall be specified for the 0.95 or greater confidence interval. The EDL shall be established initially and verified annually for each method and sample matrix.

c) The analytical detection limit (L_d) is the smallest amount of an analyte that can be distinguished in a sample by a given measurement procedure throughout a given (e.g., 0.95) confidence interval. The analytical detection limit shall be established initially and verified annually for each method and sample matrix.

c) Method Uncertainties - The laboratory shall have the ability to trace all sources of method uncertainties and their propagation to reported results.

D.4.5 Data Reduction

Procedures utilized in the computation of final concentrations of radioactive materials shall include an independently derived verification of results.

D.4.6 Quality of Standards and Reagents

A radioanalysis laboratory must have an operational internal quality control program that ensures that all radiation detection instruments are calibrated and functioning.

a) The quality control program shall establish and maintain provisions for radionuclide standards traceability.

1. Reference standards that are used in a radioanalytical laboratory must be obtained from either the National Institute of Standards and Technology (NIST) or suppliers who participate in supplying NIST standards or NIST traceable radionuclides.

2. NIST standards must be accompanied with a certificate of calibration which describes the standard's (l) principle radionuclide, mass or volume, and chemical composition; (2) reference time and date; (3) measurement result (activity of principal and possible daughter radionuclides per gram of solution; (4) measurement method; (5) a statement of purity (list of known or suspected radionuclide impurities, their activities, and how they were measured); (6) decay information (statement of the assumed half-life and other decay information); and (7) and estimate of errors (includes errors from the measurements themselves and those created by the decay assumptions).

3. In all radionuclide measurements, the volumes, shapes, and physical and chemical characteristics of the standards and their containers must be as identical as practicable for the most accurate results.

D.4.7 Selectivity

Gross measurements such as Gross alpha, beta, or gamma

measurements may be used in screening general activity levels, however they must not be used to characterize a sample.

D.4.8 Constant and Consistent Test Conditions

To prevent incorrect analysis results caused by the spread of contamination among samples, the laboratory shall establish and adhere to written procedures to minimize the possibility of cross-contamination between samples. High- and low-activity samples shall be either processed in different laboratories, or separate, distinct locations of the laboratory. Weekly surveys of gross activity levels in the laboratory shall be conducted and any detectable contamination shall be removed.

D.5 Air Testing

Analyses for Air Toxics shall follow the essential quality controls for chemistry outlined in Section 5.5.4.1. For air testing, the blank, laboratory control sample and a desorption efficiency (such as charcoal tubes) shall be used. Matrix spikes and duplicate samples shall be used when feasible.

Figure D-1

USE OF REFERENCE CULTURES (BACTERIA)
Flow Chart

Reference culture from source recognized by NELAC

Culture once
Purity Checks and Biochemical Tests as Appropriate

Reference Stocks
Retained under specific Conditions:
Freeze dried, liquid nitrogen storage, deep frozen or other storage means under specified conditions and storage times/

Purity Checks and Biochemical Tests as Appropriate

Thaw/Reconstitute
Purity Checks and Biochemical Tests as Appropriate

Working Stocks
Maintained under specific conditions and storage times

Regular/Daily Quality Controls